

THE DETERMINATION OF ORGANOPHOSPHORUS PESTICIDES IN MUNICIPAL AND INDUSTRIAL WASTEWATER BY EPA 614

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Facility Name: _____ VELAP ID: _____

Assessor Name: _____ Analyst Name: _____ Inspection Date: _____

Relevant Aspect of Standards**Method
Reference****Y****N****N/A****Comments***Records Examined:* SOP Number/ Revision/ Date _____ Analyst: _____

Sample ID: _____ Date of Sample Preparation: _____ Date of Analysis: _____

Was glassware cleaned by first rinsing with the last solvent used in it, followed by washing with hot water and detergent, and then rinsed thoroughly with tap and reagent water?

3.1.1

After cleaning, was glassware muffled at 400°C?

3.1.1

Before analyzing any samples, was a reagent water blank used to demonstrate that all glassware and reagent interferences were under control?

8.5

Each time a batch of samples was extracted or each time a reagent was changed, was a laboratory reagent processed to safeguard against contamination?

8.5

Was Sodium Sulfate either heated at 400°C for 4 hours, OR heated at 450-500°C for 16 hours, OR subjected to Soxhlet extraction with methylene chloride for 48 hours?

6.5

Was Florisil heated for at least 16 hours at 130°C prior to use?

6.8

Were samples collected in glass containers?

9.1

Were samples iced or refrigerated at 4°C from collection until extraction?

9.2

Notes/ Comments:

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Relevant Aspect of Standards	Method Reference	Y	N	N/A	Comments
External Calibration Procedure:					
Was calibration performed at a minimum of three concentration levels?	7.2.1				
Was an Average Calibration Factor used for quantitation only if the relative standard deviation of the calibration factor was less than 10% over the entire ranges?	7.2.2				
Was the calibration curve verified on each working shift by the measurement of one or more calibration standards to be no more than $\pm 10\%$ of the predicted response?	7.2.3				
Internal Calibration Procedure:					
Was calibration performed at a minimum of three concentration levels?	7.3.1				
Was an Average Response Factor used for quantitation only if the relative standard deviation of Response Factor was less than 10% over the entire working range?	7.3.2				
Was the calibration curve verified on each working shift by the measurement of one or more calibration standards to be no more than $\pm 10\%$ of the predicted response?	7.3.3				
Establish Accuracy and Precision					
Did the analyst demonstrate the ability he could produce data that met the requirements indicated in Table 2 of the reference method?	8.2.4				
Extraction					
Were samples extracted within 7 days of collection?	9.3				
Were sample extracts analyzed within 40 days of extraction?	9.3				
Notes/ Comments:					

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Relevant Aspect of Standards	Method Reference	Y	N	N/A	Comments
Was the entire sample poured into the separatory funnel with the sample container being marked so that sample volume could be determined later to nearest 5 mL?	10.1 10.8				
Was the sample container shaken for about 30 seconds with methylene chloride which was then poured into the separatory funnel?	10.2				
Was the sample shaken for 2 minutes with the above 60 mL of methylene chloride?	10.2				
Were the organic and aqueous phases allowed to separate for at least 10 minutes?	10.2				
Were the aqueous and organic phases separated and the remaining aqueous phase extracted twice more?	10.3				
Was the combined extract poured through a drying column of sodium sulfate?	10.5				
Was the extract condensed to about approximately 1 mL on K-D apparatus using a Snyder column?	10.6				
Optional Cleanup: Acetonitrile Partition					
If an alternative cleanup procedure other than the 3 described in the reference method was used, the recovery of each compound of interest must be confirmed to be at least 85%?	11.1				
Was the concentrated sample extract first combined with hexane to reach about 15 mL volume?	11.2.1				
Was this volume then extracted 4 times with 30-mL portions of hexane-saturated acetonitrile by shaking vigorously for 1 minute?	11.2.1				
Was 650 mL of reagent water and 40 mL of saturated sodium chloride solution then added to the combined acetonitrile phases and mixed thoroughly?	11.2.2				
Was the resultant mixture extracted 2 times in a separatory funnel by shaking with 100 mL portions of hexane?	11.2.2				
Notes/ Comments:					

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Relevant Aspect of Standards	Method Reference	Y	N	N/A	Comments
Were the combined hexane extracts poured through a drying column of sodium sulfate and condensed in a K-D apparatus?	11.2.4				
Optional Cleanup: Florisil Cleanup					
Was a column prepared that contained prepared florisil with prepared sodium sulfate on top?	11.3.1				
Was the column rinsed with hexane?	11.3.1				
Was the column eluted 3 times with ethyl ether in hexane into 3 separate K-D flasks?	11.3.3				
Were the eluates concentrated in a K-D apparatus?	11.3.4				
Optional Cleanup: Removal of Sulfur					
If sulfur was present in extracts, was this technique used to cleanup the Florisil-extracted fractions?	11.4				
Was a column prepared with prepared alumina underneath prepared sodium sulfate?	11.4.3				
Was the sample extract rinsed through the column with hexane?	11.4.3				
Quality Control					
Were matrix spikes analyzed at a frequency of 10% of all samples or at least one per month, whichever was greater?	8.1.3 8.4				
Were matrix spikes assessed against criteria of ± 3 Standard Deviations?	8.3				
If a matrix spike recovery did not meet the criteria, were all of the samples analyzed in association qualified? [NOTE: This is stricter than NELAC Appendix D Indications]	8.4				
When doubt existed over the identification of a peak, was a confirmatory technique such as a dissimilar column, specific element detector, or mass spec. (see section 14) used?	8.6				
Notes/ Comments:					

 **Table 2.** Single-Operator Accuracy and Precision

<i>Parameter</i>	<i>Average Percent Recovery</i>	<i>Standard Deviation (%)</i>	<i>Spike Range (µg/L)</i>	<i>Number of Analyses</i>	<i>Matrix Types</i>
Diazinon	94	5.2	0.04–40	27	4
Parathion methyl	95	3.2	0.06–60	27	4
Parathion ethyl	102	4.1	0.07–70	27	4